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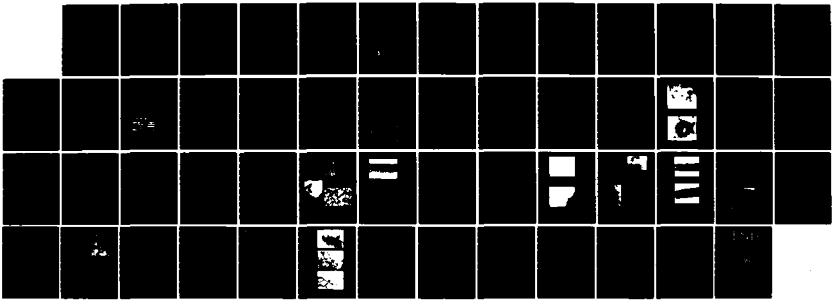

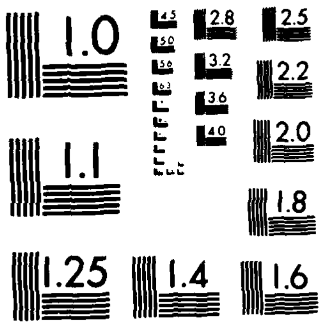

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RAPID SOLIDIFICATION PROCESSING OF MAGNESIUM ALLOYS

September 1984

M. C. FLEMINGS and A. MORTENSEN
Department of Materials Science & Engineering
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

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ABSTRACT

This report comprises the second year of a two year program on Rapid Solidification, extrusion processing and mechanical property measurement of Magnesium alloys.

During the first year, the Rapid Solidification of various Mg alloys and the resultant structures were investigated in two stages. The first involved the study of the Mg-Zn system processed by chill casting and melt spinning. It was observed that the amount of second phase decreased with decreasing distance from the chill surface to eventually become completely suppressed in a featureless zone. The second stage dealt with the splat quenching and melt spinning of Mg-5%Zn, Mg-8%Al, EZ33, AZ91, and QE22. In all the alloys, the presence of a featureless zone was observed that was shown in the Mg-Zn system to consist of a homogeneous solid solution having the initial alloy composition. For the Mg-5%Zn, EZ33, and ZE41 alloys, melt spun ribbons were almost completely featureless throughout a 70 μm average ribbon thickness. Microhardness measurements were taken for alloys EZ33 and ZE41, and the rapidly solidified featureless regions were found to have a greater hardness than both the as-received (primary phase) alloys and the rapidly solidified dendritic structures.

During the second year, samples were cast using a controlled atmosphere (He gas), and vertical wheel melt-spinning apparatus contructed at MIT. Casting conditions were thus perfected, to obtain larger quantities of ribbons of consistent quality. Extrusion procedures were investigated on rapidly solidified magnesium alloys. A gradual lowering of the extrusion temperature from 470°C to 210°C yielded improved mechanical properties on alloys ZK60, EZ33, and Mg-5.3%Zn. Optimal conditions were: a reduction in area ratio of about 1/30, and an extrusion temperature of about 200-250°C. Results on alloy ZK60, extruded under these conditions, displayed interesting mechanical properties, and made it comparable in specific properties to R.S.P. aluminum alloys.

Tensile properties in the as extruded condition were: Y.S. 53.0 ksi, and U.T.S. 56.3 ksi. The ductility in particular was significantly higher than that of wrought ZK60 alloy: 19.6% (typical mechanical properties of the conventional extruded material are: Y.S. 41 ksi, U.T.S. 51 ksi, elongation, 11%). Originator furnished key

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FOREWORD

This final technical report covers the activity performed under the U.S. Army Contract DAAG-46-82-K-0051 for "Rapid Solidification Processing of Magnesium Alloys" during the period of August 1, 1982 - July 31, 1983 with Professor Merton C. Flemings, Department of Materials Science & Engineering, Massachusetts Institute of Technology as principal investigator. The contract was administered under the direction of the Army Materials & Mechanics Research Center, Watertown, MA 02172, with Dr. Judith J. Kohatsu as Program Technical Monitor.

FIGURES

TABLES

Abstract

This report comprises the second year of a two year program on Rapid Solidification, extrusion processing and mechanical property measurement of Magnesium alloys.

During the first year, the Rapid Solidification of various Mg alloys and the resultant structures were investigated in two stages. The first involved the study of the Mg-Zn system processed by chill casting and melt spinning. It was observed that the amount of second phase decreased with decreasing distance from the chill surface to eventually become completely suppressed in a featureless zone. The second stage dealt with the splat quenching and melt spinning of Mg-5%Zn, Mg-8%Al, EZ33, AZ91, and QE22. In all the alloys, the presence of a featureless zone was observed that was shown in the Mg-Zn system to consist of a homogeneous solid solution having the initial alloy composition. For the Mg-5%Zn, EZ33, and ZE41 alloys, melt spun ribbons were almost completely featureless throughout a 70 um average ribbon thickness. Microhardness measurements were taken for alloys EZ33 and ZE41, and the rapidly solidified featureless regions were found to have a greater hardness than both the as-received (primary phase) alloys and the rapidly solidified dendritic structures.

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I. INTRODUCTION

Rapid Solidification Processing (R.S.P.), the subject of numerous research projects in Materials Science and Engineering for the past 20 years, has led to the obtention of considerable mechanical property improvements in a number of alloys. Possible, or existing applications of R.S.P. metals are many, ranging from turbine blades[2] to magnetic recording heads[3]. Alloys of aluminum have received considerable attention, and research on those has yielded numerous payoffs in terms of improved mechanical properties, as was exposed in our previous report[1].

Magnesium alloys, on the other hand, have so far barely been explored in that light. It is the ambition of this project to help fill in that gap, and explore the processing and the engineering porperties of R.S.P. magnesium alloys.

Published articles dealing with the subject are relatively few. In one article, the nature and the mixing enthalpies of intermediate and Al-rich phases of the Al-Mg system were investigated using a rapid solidification technique[4]. Metastable solid solutions or intermatellic compounds were found to exist. In another article[5], the increase in solubility in magnesium of

1. Rapid Solidification Processing, Final Technical Report, 1981-1982, Contract Number DAAG 46-81-K-0051.
2. Rapid Solidification Processing: Principles and Technologies II, Ed., R. Mehrabian, B.H. Kear, and M. Cohen, Claitor's Publishing Division, Baton Rouge, 1980, page 1.
3. T. Masumoto, Proc. 4th Int. Conf. on Rapidly Quenched Metals, Sendai 1981, page 5.
4. B. Predel and K. Hulse, Z. fur Metallkunde, 69, page 690, 1978.
5. N.J. Varich and B.N. Litvin, The Physics of Metals and Metallography, 16, No. 4, 1968, page 29.

Mn or Zr after splat cooling was explored using lattice parameter measurements obtained by X-ray diffraction. The thermal stability of the supersaturated Mg-Mn phases was also explored. In particular, an increase in the solubility of Zr in Mg to a value of 1.2 wt % is claimed by splat quenching "small portions of the melt (at above 800 C° onto a copper chill)". However, this seems to contradict data given by Emley[6] on an already confused issue.

The Mg-Zn system has been found to form metallic glasses by melt-spinning alloys in the range from $Mg_{80}Zn_{20}$ to $Mg_{60}Zn_{40}$[7]. Crystallization and recrystallization kinetics of amorphous alloys in that range were studied. Electron transport properties have also been investigated on these metallic glasses[8].

Work on densified products emanates mainly from the closely related powder metallurgy field. Although in one instance, the standard compaction-sintering path was used[9], most often extrusion was preferred as a means of densification. The advantage is to be found in the far greater amounts of shear deformation involved in the latter process, an aspect of importance in breaking off the oxide layer surrounding the magnesium particles, to promote metal-metal bonding. Magnesium powder extrusions, alloy extrusions, or powder mixture extrusions

6. E.F. Emley, Principles of Magnesium Technology, Pergamon Press, 1966, page 254.
7. Z. Altounian, et al., J. of Materials Science 17,1982, page 3268.
8. T. Matsuda and U. Mizutani, J. Phys F: Met. Phys., 12, 1982, page 1877.
9. S. Storchheim, Int. J. of Powder Metallurgy, 8, (3), 1982, page 115.

have been reported by Emley[10], Foerster[11], Fischer[12], and Isserow and Rizzitano[13]. Pure Mg metal powders have been extruded, to investigate to what extent the oxide could produce properties similar to those in S.A.P. aluminum. Some results are given in Table I. A variety of processes such as interference hardening, first described by Busk and Leontis[14] were invented and explored. Work on pre-alloyed quenched powders is relatively rare, however. Busk and Leontis hot extruded atomized ZK60 powders, as well as alloys AZ31, M1 and AMZ111, the three first alloys being commercial. ZK60 gave the best results, as shown in Table II and was even produced on a commercial basis[10] as (P) ZK60B. Isserow and Rizzitano[13] also extruded ZK60, starting with powders obtained by the rotating electrode process. Here the powders were sealed in an aluminum container, and evacuated prior to extrusion. Extrusion temperatures varied from ambient to 370°C (700°F). Results are given in Table II and display excellent mechanical properties. But fracture surfaces show that the products delaminated and care must be exerted in judging the observed longitudinal properties, as they may be highly inferior in directions orthogonally to the extrusion direction. Extrusion reduction in area ratios were 1/10. Also, Storchheim extruded atomized Mg-1% Zn-Si powders, with the results shown in Figure 1.

Magnesium alloys are generally not quite as strong as those of aluminum, on an equal volume basis, but are on the other hand, much lighter. If we compare the specific properties of the two metal alloys, that is the ratio strength/density; to achieve the same specific strength as a typical R.S.P. alluminum alloy with a yield strength of 90 ksi (a reasonable value, as can be seen in

10. E.F. Emley, Supra note 2, page 531.
11. G.S. Foerster, Metals Eng. Quarterly, 12, (1), page 22.
12. P.A. Fisher, Int. Metals Reviews, 1978, No. 6, page 269.
13. S. Isserow and F.J. Rizzitano, Int. J. of Powder Metallurgy, 10, (3), July 1974, page 217.
14. R.S. Busk and T.E. Leontis, Trans. AIME, Vol. 188, Feb. 1950, J. of Metals, page 297.

Typical Screen Analysis and Packing Densities of Powders Used

Grade	Packing density g/cm³	Approx. B.S. screen analysis (%)				Remarks
		44–100	72–100	100–240	Passing 240	
Grade 2A	0·4	71	–	28	0·3	
3	0·7	–	0·2	98	2	Ball milled to increase density
4	0·4	–	3	89	8	
4F		–	–	–	100	Fines obtained by sieving through 240 BS

Comparative Properties of Conventional and Powder Extrusions in Pure Magnesium ($\frac{3}{8}$ in. rod from 3 in. Container)

Material extruded	Extrusion conditions		Tensile properties of extrusion		
	Temp. (°C)	Speed (ft/min)	0·1 P.S. (tsi)	U.T.S. (tsi)	El. (%)
Cast billet	420	15	4·6	12·4	9 (on 1 in.)
Powder 4	420	30	15·2	22·7	3½
Powder 4F	450	75	17·9	20·6	4 (on 4√ area)
Powder 4F + Be	455	120	21·0	22·6	5½

From Ref 6 .

Properties of Dispersion-Hardened Magnesium Extrusions

Composition	Fabrication Data	Extrusion* Shape	Extrusion* Temperature, °F	Extrusion* Speed, fpm	Elongation, Pct	Yield Strength, psi Tension	Yield Strength, psi Compression	T Str
Mg	<1 μ powder	$\frac{1}{8}$ in. diam.	1100	<1	1	35,000	36,000	4
ZK10 + 2MgO	Screw extruded	$\frac{3}{8}$ by $\frac{3}{4}$ in	820	4	7	35,000	22,000	4
Mg-1 Zn-1.6 Si	Atomized, 35/65 mesh	$\frac{1}{16}$ by $\frac{3}{4}$ in.	600	5	3	43,000	36,000	5
Mg-1 Zn-1.6 Si	Atomized, 35/65 mesh	$\frac{1}{16}$ by $\frac{3}{4}$ in.	600	100	7	34,000	22,000	4
Mg-1 Zn-1.6 Si	Atomized, −100 mesh	$\frac{1}{16}$ by $\frac{3}{4}$ in	600	100	4	45,000	29,000	4
AK11	Interference hardened†	$\frac{1}{16}$ by $\frac{3}{4}$ in.	600	100	8	44,000	45,000	5
AK11	Interference hardened†	$\frac{1}{16}$ by $\frac{3}{8}$ in.	900	70	10	48,000	34,000	5

*$\frac{1}{8}$ in. diam. wire was extruded from $\frac{3}{4}$ in. container, others, from 3 in. container.

†Atomized magnesium-zirconium pellets were coated with aluminum flake powder, compacted at 650°F and heat treated 16 hr at 780°F.

From Ref 11 .

TABLE I . Properties of Magnesium Powder Extrusions .

Comparison of As Extruded Properties of Alloys Extruded from Powder and from Permanent Mold Billets

Shape	Extrusion Conditions		Powder				Billet			
	Temp. °F	Speed Fpm	Pct E	1000 psi TTS	1000 psi CTS	1000 psi TS	Pct E	1000 psi TTS	1000 psi CTS	1000 psi TS
AZ31										
Rod	600	5	15	39	40	47	16	34	30	44
	700	5	15	36	36	45	15	34	25	45
	800	5	15	35	30	45	14	34	22	44
Strip	650	5	12	29	29	43	18	23	18	39
	650	15	12	25	18	39	16	25	15	38
	650	30	11	25	15	37	15	25	15	38
M1										
Rod	600	5	14	44	38	48	25	27	23	39
	700	5	11	43	35	47	23	30	24	40
	800	5	13	43	28	47	14	33	19	43
Strip	650	5	6	34	33	43	23	16	16	23
	650	15	9	30	26	40	19	18	14	35
	650	30	8	28	19	40	18	18	14	35
AMZ111										
Rod	600	5	12	40	41	48	19	37	31	45
	700	5	15	40	37	46	13	33	20	42
	800	5	12	38	34	46	11	32	16	40
Strip	850	5	5	37	36	45	10	29	19	40
	650	20	9	33	31	42	8	29	13	38
	650	40	8	32	24	39	9	28	13	38
ZK60										
Rod	600	5	16	44	48	53	17	41	41	54
	700	5	17	41	43	51	14	40	34	51
	800	5	15	37	38	48	15	39	27	48
Strip	850	5	10	43	43	52	10	40	40	52
	650	20	6	37	37	47	13	38	36	49
	850	40	7	35	30	45		Hot Short		

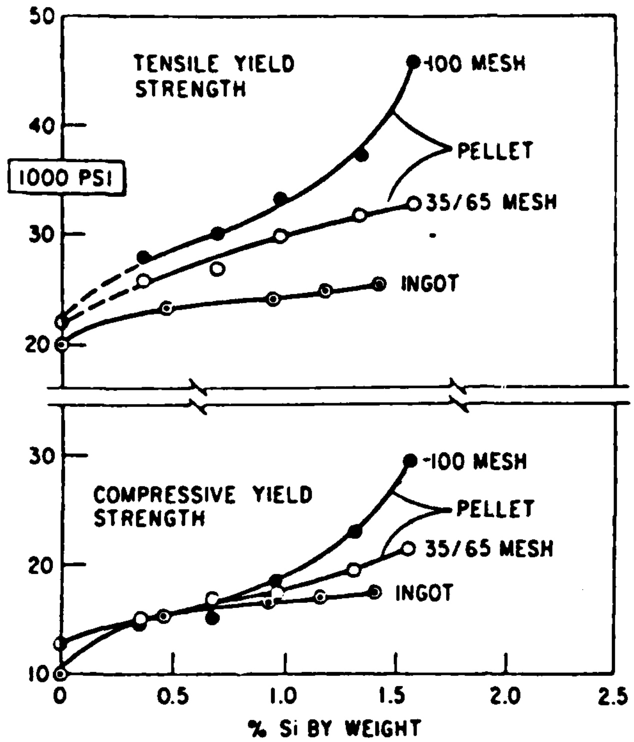
TABLE II - Properties of extruded Mg Alloys . Ref 14 .

Mechanical Properties of Magnesium ZK60A Rods from Microquenched Powders

Identification	Ext. temp °F	Aging temp °F (24 hr)	0.2 Y.S. ksi	U.T.S. ksi	El. %	RA %	Charpy V-notch ft.-lb.
Commercial	700	300	40.5	49 1	21.0	44.6	5.7
Microquenched							
K-1*	250	none	48.0		25		
		250	51.4		23		30
K-2**	RT	none	56.5		6		
		250	61.4		17		20; 29
D**	RT	none	54.0	58.5	4.0	5.0	
		250	53.1	57.9	19.0	41.0	
		300	57.2	61.2	4.0	2.6	
E**	150	none	54.5	56.5	17.0	28.1	
		250	57.5	61.0	14.0	32.2	18.5
		300	53.5	59.5	14.0	35.4	15.8

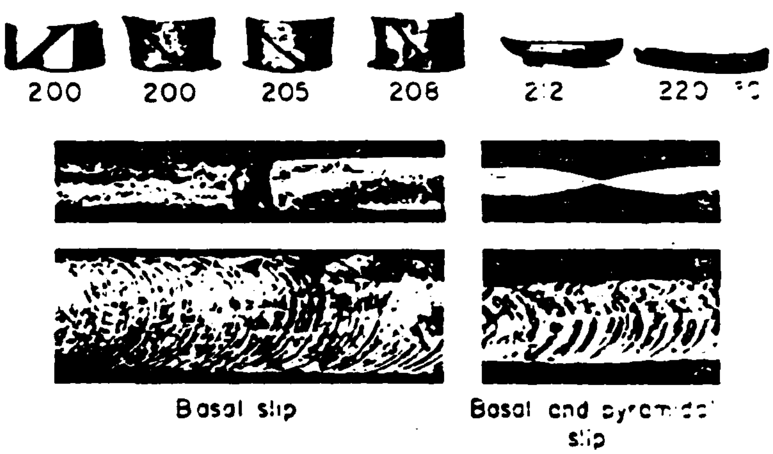
* Air-cooled after extrusion.
** Water-quenched after extrusion.

Table III - Properties of Microquenched ZK 60 . Ref 13 .



—Properties of Mg-1 Zn-Si extrusions.

Figure 1 - From Ref 9 .



(a) Sudden change in the plastic properties of a magnesium alloy, AZM, with increasing temperature.

(b) Slip bands in cold and hot stretched magnesium crystals.

Figure 2 - From Ref 16 .

our previous report), an R.S.P. magnesium alloy must attain a yield strength of about (90 x 108/166) = 58.5 ksi. Such mechanical properties seem perfectly feasible in magnesium alloys by R.S.P., and are indeed approached on one sample in this project. Such alloys could be of definite interest in aerospace applications, where specific properties are important.

Magnesium has a hexagonal crystal structure, as do most of its solid solutions. An exception is obtained when more than 10% Lithium is alloyed to magnesium, where the structure becomes cubic [15]. At room temperature, only one slip system, operating on the basal (0001) planes is active, together with twinning, to allow plastic deformation of the metal. Formability is therefore poor at room temperature. For each alloy, however, there is a temperature slightly above 200°C, at which pyramidal slip planes become active, with drastic increases in formability[16]. That temperature is 225°C in pure magnesium, and the change is illustrated in Figure 2.

Heat treating temperatures lie usually in the range 150-250°C for aging, see Table IV. Moreover, melt-spun ribbons of EZ33 and ZE41 showed no decrease in hardness properties when treated to the T5 condition at 180°C, for 12 and 16 hours respectively[1].

In this work, magnesium alloys have been rapidly solidified by melt spinning small quantities onto a rotating copper wheel. The ribbons were subsequently consolidated by first compacting them, and then extruding the green compact. For this last step, there is a compromise to be obtained, between the need for a high temperature, above 220-225°C for improved formability, and a low temperature necessary to retain the quenched-in structures, or interesting modifications thereof, below about 250 C, as was

15. E.F. Emley, Supra note 2, page 299.
16. E.F. Emley, Supra note 2, page 483.

Table IV - From Ref .6 .

TYPICAL HEAT TREATMENT SCHEDULES FOR CASTING ALLOYS

Type of treatment	Alloy	Conditions		Typical Tensile Properties†						US Treatment (where difference appreciable)		Remarks
				As cast			Heat treated					
		Time (hr)	Temp. (°C)	0·1% P.S.	U.T.S.	% El.	0·1% P.S.	U.T.S.	% El.	Time (hr)	Temp. (°C)	
Annealing	ZRE1	10–16	170–180	5½	10	4	5½	10	4	{ 2 6	343 215	§
	ZT1	16	315	4½	13½	13	5½	15	7			
Precipitation	Z5Z	10–16‡‡	170–180	7¾	15	10	9½	17	7			
	RZ5	(a) { 2 10–16 (b) 24	330 170–180 250	6	11½	5	8½	14	4			
	TZ6	{ 2 10–16	330 170–180	7¾	17	17	10	17½	10			
Solution	A8	{ 8 16	380–390 410–420	5	10	4	4¾	16	13	(a) 18 (b)†= { 6 2 10	410 410 350 410	††
Full, with air cooling	AZ91	{ 8 16 10	380–390 410–420 200	5½	10	2	5½ 7½	15 15½	7 (ST) 2 (FHT)	{ 18 16 †= { 6 2 10	410 170 410 350 410	††
	AZ92	(a) { 18 16 (b)†= { 6 2 10 16	405 170 405 350 405 270	((14))	(24)	–	((22))	(40)	–			††
	AZG (AZ63)	{ 12 5	385 220	((14))	(29)	–	((19))	(40)	–			††
	HK31	{ 2 16	565 200	4½	9	5	6½	14	7			=
	Z6Z (ZK61)	{ 2 48	500 130	((22))	(38)	–	((28))	(43)	–			
Full, with liquid quenching	MSR	{ 4–8 Quench 8–16	520–530 200	6½	11½	7½	12	16½	5			§§

§ The 2-stage treatment is not recommended since heating at 343°C (650°F) reduces creep resistance.

† Tensile properties not enclosed in parentheses are expressed in tsi; these figures relate to British D.T.D. test bars. ( ) denotes figures expressed in kpsi; these results relate to ASTM bars. (( )) denotes 0·2 per cent PS values expressed in kpsi; these results relate to ASTM bars. Multi-stage treatments are bracketed; alternative treatments are marked (a), (b) etc.

‡ Castings to be loaded in furnace at operating temperature.

‡‡ Optionally preceded by 2 hr at 330°C; this effects stress relief.

†† U.S. practice is to load castings into furnace at about 250°C and raise furnace to operating temperature over about 2 hr.

ST – Solution heat treated. FHT = Fully heat treated.

§§ Few per cent SO_2 in furnace atmosphere necessary to minimize formation of RE and Zr hydrides with reduction in tensiles.

†‡ Alternative treatment designed to prevent grain growth.

explained above. Usual extrusion temperatures for magnesium lie in the range 300-450°C[17,18].

In what follows, three alloys are examined: EZ33, azirconium grain-refined cast alloy containing zinc and rare earth; ZK60, a high strength wrought alloy containing 5.5% zinc, also grain-refined with zirconium, and a binary 5.3% zinc alloy, cast at MIT. The processing steps named above are summarized in Figure 3, the double extrusion procedure having been used on one sample, of alloy ZK60.

17. E.F. Emley, Supra note 2, page 544.
18. "Extrusion," by K. Laue, H. Stenger; American Society for Metals, 1981.

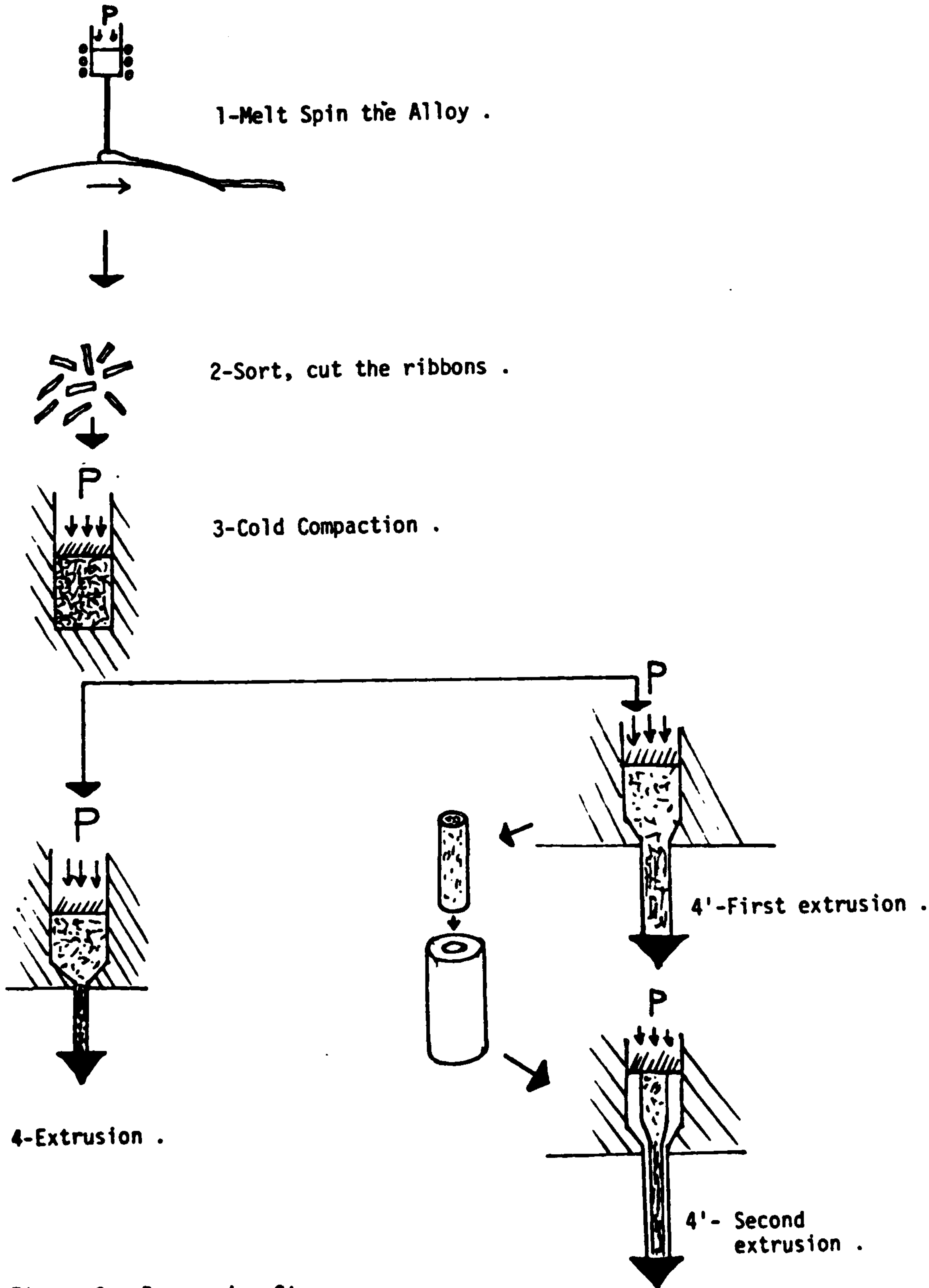


Figure 3 - Processing Steps .

II. EXPERIMENTAL PROCEDURES

A. Alloying - Alloys EZ33 and ZK60 were both purchased as such. Mg-Zn binary alloys were prepared at MIT, in a gas furnace, using a steel crucible and standard melting and alloying procedures with Dow flux #310[6]. All castings were analyzed for Zn by atomic absorption, and results are given in Table V. Nominal compositions were selected to be close to those of the commercial Zn-Zr containing wrought alloys ZK60 and ZK20, and otherwise scan by increments of 2% the low Zn containing compositions. Ingots cast were about 1.5 kg in mass. Initial metals were 99.9% pure Mg and analytical Zn.

B. Melt-Spinning Procedures - Two melt-spinning apparati were used. The first is described in the previous report, and was used until a more performant apparatus was built within the Solidification Processing Group at MIT. Alloy EZ33, and pure magnesium were cast on this first melt-spinner.

The second wheel casting apparatus (Figures 4, 5, 6, and 7) allows for single roll strip casting on the outside edge of a rotating wheel; the entire melt spinner is housed inside a vacuum-tight enclosed chamber. This permits strip casting experiments to be conducted in vacuum or under controlled atmospheres (He gas). The casting substrate is a wheel of 14 cm diameter and 3.8 cm width made of OFHC copper. The wheel is mounted on the shaft of a "Ferromatic" rotary vacuum feedthrough. The wheel has been precisely balanced on the shaft to insure smooth rotation at high speeds. The wheel is driven by a 1/7 H.P. variable speed, series wound motor capable of 10,000 rpm, which gives a substrate speed of up to 73 m/sec. The shaft of the motor is linked by a flexible coupling to the rotary feedthrough shaft. This minimizes the motor vibrations transmitted to the wheel via the drive shafts.

TABLE V

Binary Mg-Zn Alloys Cast at MIT.

Ingot #53	2.5% Zn.
Ingot #62	4.3% Zn.
Ingot #38	5.3% Zn.
Ingot #41	5.3% Zn.
Ingot #61	6.4% Zn.

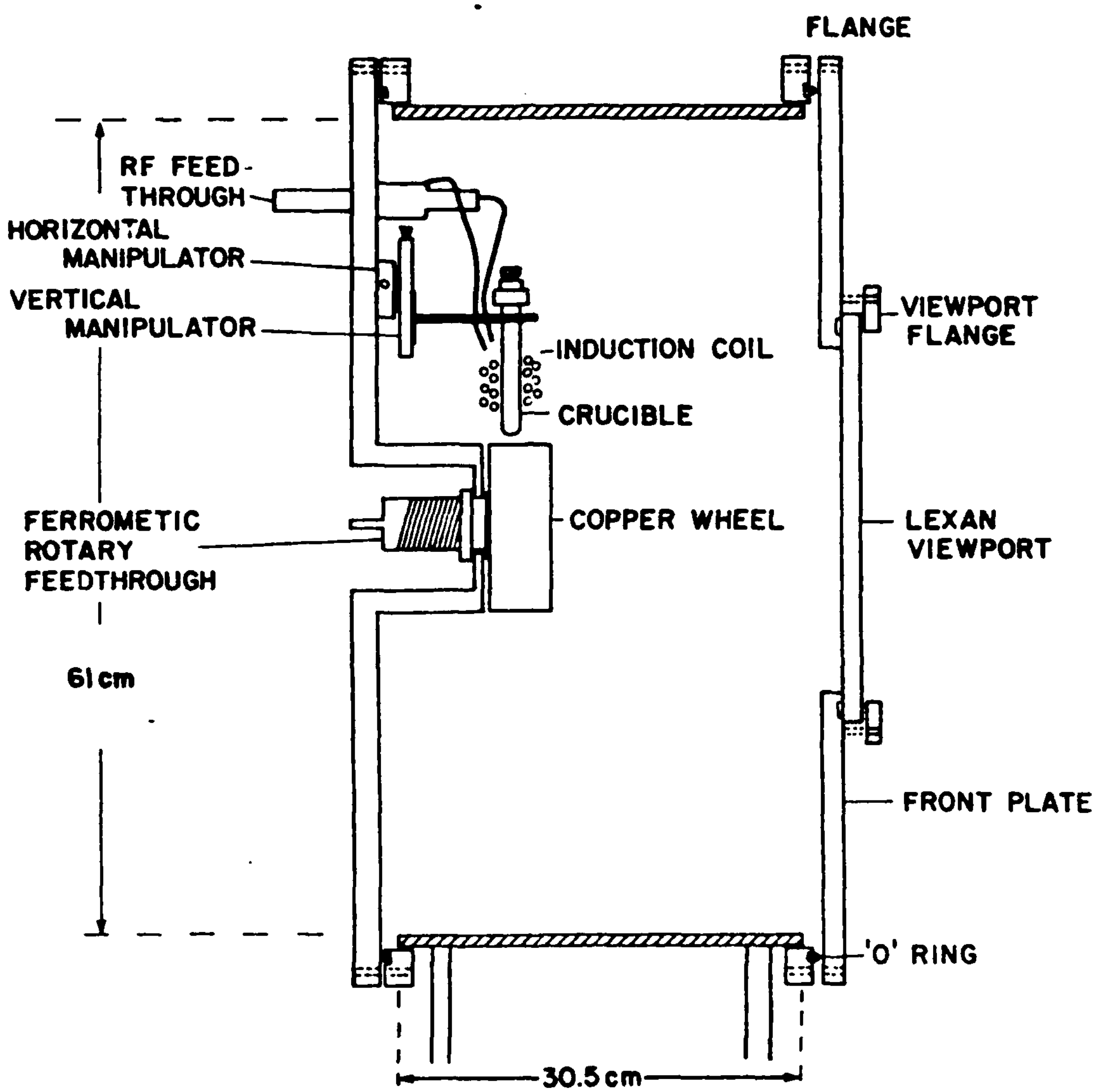


Figure 4 - Schematic illustration of strip casting apparatus, side view.

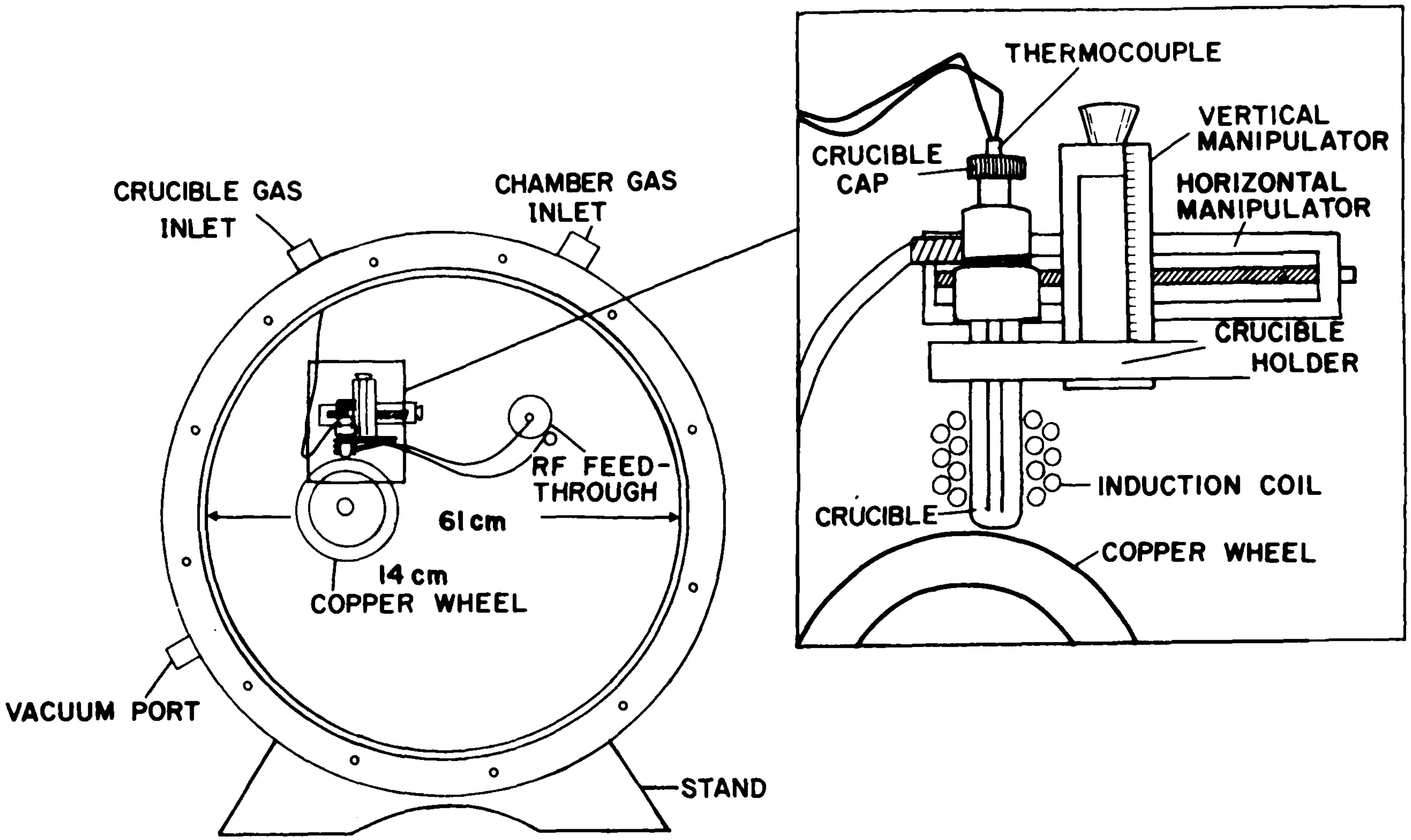


Figure 5. Schematic illustration of strip casting apparatus, front view.

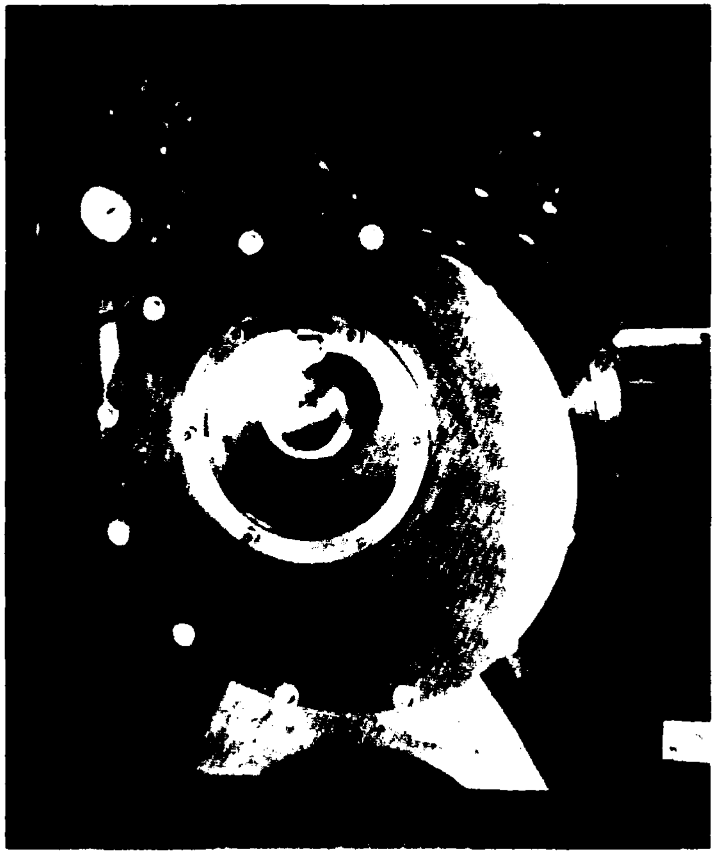


Figure 6. Strip casting apparatus.

Figure 7. Strip casting apparatus, wheel and crucible assembly.

The charges are induction heated by a 10KHZ, 20KW inverter power supply. The mild steel crucible is surrounded by an alumina holder, onto which a brass cap is attached. The brass cap allows a pressurized gas to be fed over the molten charge to force it over the moving copper wheel.

The cap also has a feedthrough, allowing a thermocouple covered by a mild steel sheath to measure the temperature of the melt. Runs in this apparatus were conducted under a helium atmosphere, thus suppressing any burning of the molten magnesium, and also minimizing boundary layer effects at the wheel[19].

The alloys were cast at 700°C, and to minimize metal evaporation, heat-up times were short, around 3 minutes. The gas ejection pressure was 40 KPa, and substrate velocities around 35 m/sec. The ejection hole diameter was about 0.5 mm (drill #74). The ribbons produced had a thickness that varied around 50 um and a width of about 3 mm.

One considerable source of problems was the fact that zinc containing alloys wet iron extremely well - which makes sense when remembering that zinc wets iron so well that it is used in plating. This fact caused the molten metal jet to be very unstable, and to form large droplets that would stick to the crucible's outer side, as depicted in Figure 8a. To solve this problem, a coating of MgO mold wash was applied to the tip of the crucible, and nice ribbons were obtained, Figure 8b.

Ribbon formation was always accompanied by some splatter, that was reduced, but never suppressed. Two available set-ups were used, one able to cast samples of about 4 grams, the other casting about 20 grams at a time, thus reducing the number of runs necessary for one extrusion from 5-10 to one or two.

19. H. Liebermann, "Rapidly Quenched Metals III," 1978, Ed., B.Cantor, The Metals Society.

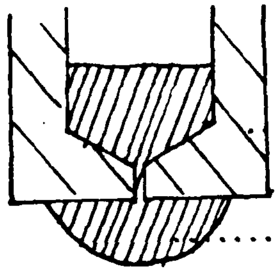


Figure 8-a . Uncoated crucible .

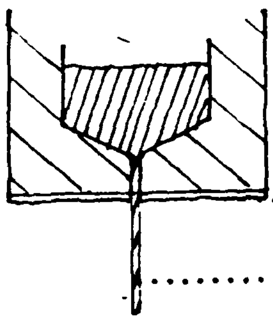


Figure 8-b , Coated crucible .

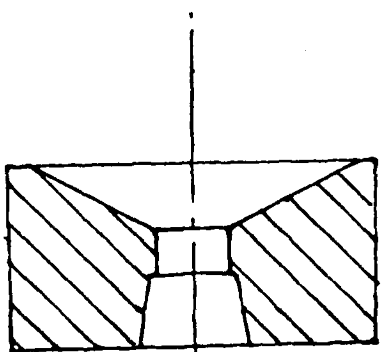


Figure 9-a . Conical Extrusion Die Geometry .

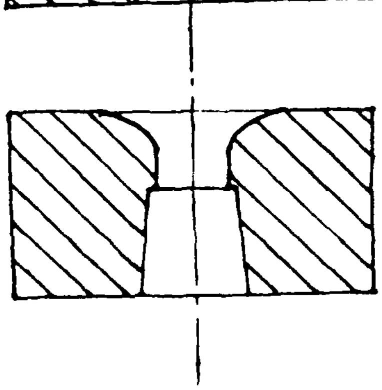


Figure 9-b . Straight Extrusion Die Geometry .

C. Densification - The apparatus was a small scale extrusion set up, built and used at MIT under a 10 ton press at first, and a 20 ton press when the need for higher loads became apparent.

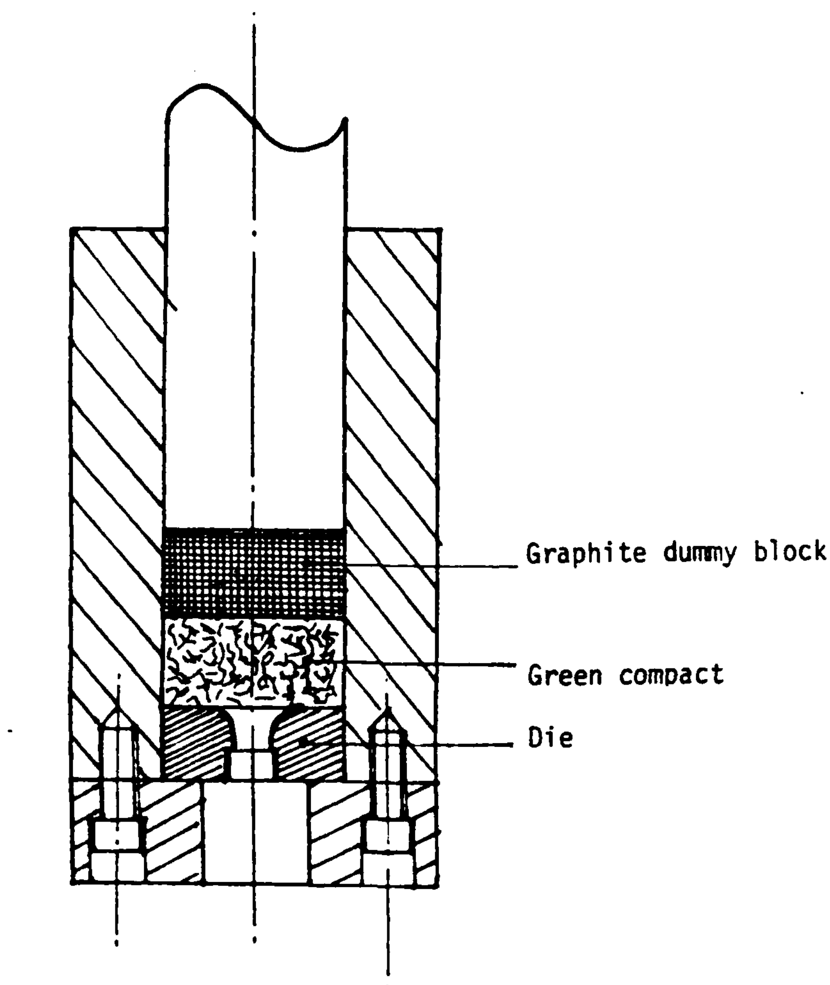
A sketch describes the set up in Figure 10. All parts were machined from stainless steel, except where otherwise indicated. Dies are easily interchangeable, and two different geometries were employed. One consists of a 120° conical die (Figure 9A), the other of a flat die with a rounded off edge (Figure 9b). The former was used to minimize losses in the extrusion butt, the second is the standard shape used in hot extruding magnesium[18].

No substantial difference in the performance of the two geometries was noticed, and they therefore were both used. The graphite dummy block was included to avoid damaging the piston on the die at the end of the extrusion runs, as well as to minimize the risk of piston seizure in the container by back extruded metal. A new dummy block was used for each run.

As described in the introduction, the densification of the ribbons involved two steps:

1. A cold compaction in the extrusion set up, under a pressure of 10,000 psi to 20,000 psi.
2. A hot extrusion of the compact, usually using the maximum load of the press, to keep the temperature at a minimum. No container was used at this stage of the project, at the risk of some gas entrapment. It should be noted, however, that successful hot extrusions were obtained by Busk and Leontis[14] without any such canning or evacuation.

The main problem to be overcome in densifying powders of metals such as magnesium or aluminum, is the necessity to shear off the oxide layer covering each particle. If this condition has not been fulfilled, the extruded product will delaminate, and produce fracture surfaces where each individual particle is quite visible and separated from its neighbor. A simple test for an



Scale 1 .

Figure 10 - Extrusion Tooling .

extruded bar consists therefore of breaking it by hand and looking at the fracture surface. It was thus found that an extrusion ratio of about 1/30 is sufficient to avoid delamination of the densified melt-spun ribbons. Reduction in area ratios of the order of 1/5 to 1/10 were on the other hand insufficient.

Again, the pressure was fixed by the capacity of the press, so the temperature varied from sample to sample. The extrusion speed was not controlled, but was low because the temperature was kept at a minimum. The surface finish of the samples was smooth and regular. As is standard practice for hot extruding magnesium, graphite was used for lubrication[18].

The initial green compact was 1 inch in diameter, and the final extruded R.S.P. alloy 3/16 of an inch in diameter, unless otherwise noted. This corresponds to a reduction in area of $(3/16)^2 = 1/28.4$. Extruded rod lengths varied between 15 and 40 centimeters.

D. Metallography - Standard techniques were used, involving alumina powder polishing, and an optical microscope. A glycol etch was used on all samples.

E. Tensile Property Evaluation

Tensile specimens were machined from the extruded rods according to ASTM specifications. Gage lengths were four times the gage diameter. The latter was 0.125 inches when allowed, less when necessary. Given the small grain size of the extrusions, this is an amply sufficient diameter. The gage length was polished smooth with a 0.3 um Al_2O_3 powder suspension. The specimens were gripped in an Instron testing machine, and tested at cross head speeds of 0.02 inches/min.

III. RESULTS - DISCUSSION

A. Pure Magnesium - One sample of compacted and extruded melt-spun ribbons of pure magnesium was produced to obtain a reference point against which the reinforcing action of alloying elements can be compared, at an equal level of oxide inclusion. Oxide inclusions are of importance in that they can modify the mechanical properties of the consolidated product, as can be seen in Table I. A similar effect is obtained on S.A.P. aluminum parts. Properties obtained are given in Table VI, together with extrusion conditions. As can be seen, by comparing with data in Table I, the reinforcing effect of the oxide is slight with these relatively coarse ribbons.

B. EZ33 - Alloy EZ33 was hot extruded and tested. The extrusion temperature was high: 470°C. The microstructure of the ribbons was similar to those given in the previous report[1]. White "Featureless" zones are apparent. Hardness properties were promising, which conditioned the choice of this alloy as a first test. The microstructure of the extruded sample is given in Figure 11. As can be seen, thanks to the slightly shifting focus range, the grain size is small, and many small precipitates are present and evenly distributed. The fracture surface, given in Figure 12, is typically ductile, and no trace of delamination is present. Thus, the extrusion ratio of 1/28.4 is clearly adequate for densification.

C. Magnesium-Zinc Alloys - Samples were cast from #41 (see Table V), and thus have a zinc content close to that of AK60. In this way, the effect of zirconium additions can be judged, as will be done in the next section.

Microstructures can be seen in Figure 13. Ribbons were thin, and abundantly display the "Featureless" zone mentioned in the previous report.

TABLE VI

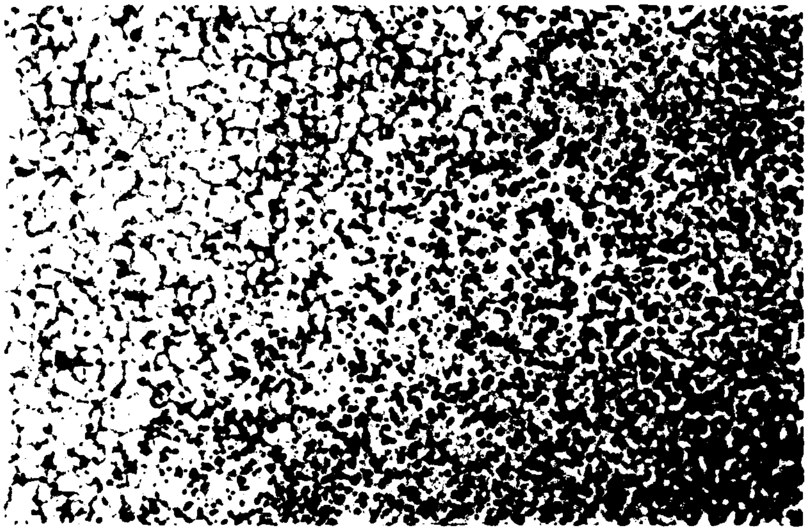
Pure Magnesium Extrusion

Extrusion Ratio	1/28.4
Extrusion Pressure	20 ksi
Extrusion Temperature	45°C 10°C
Yield Strength	22.8 ksi
U.T.S.	32.9 ksi
Elongation	11.7%

TABLE VII

EZ33 Extrusion

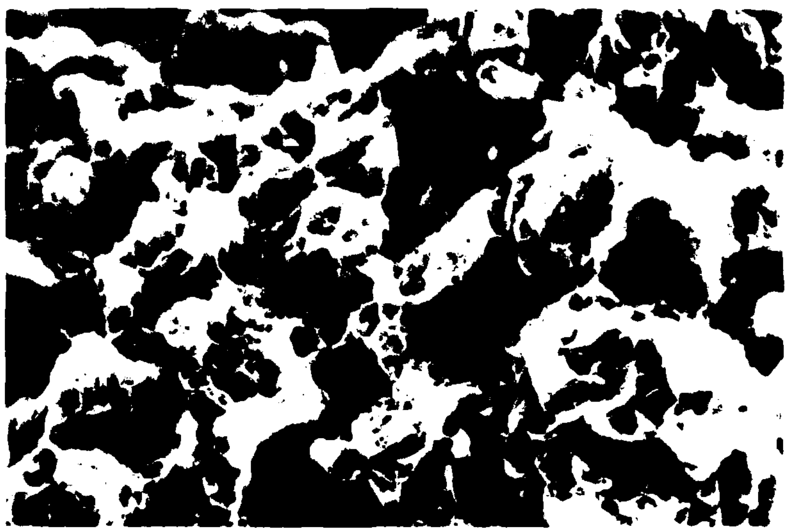
		Nominal (as cast)
Extrusion Ratio	1/28.4	
Extrusion Pressure	20 ksi	
Extrusion Temperature	470°C 20°C	
Yield Strength	32.9 ksi	16 ksi
U.T.S.	41.3 ksi	23 ksi
Elongation	14%	3%



x 1000

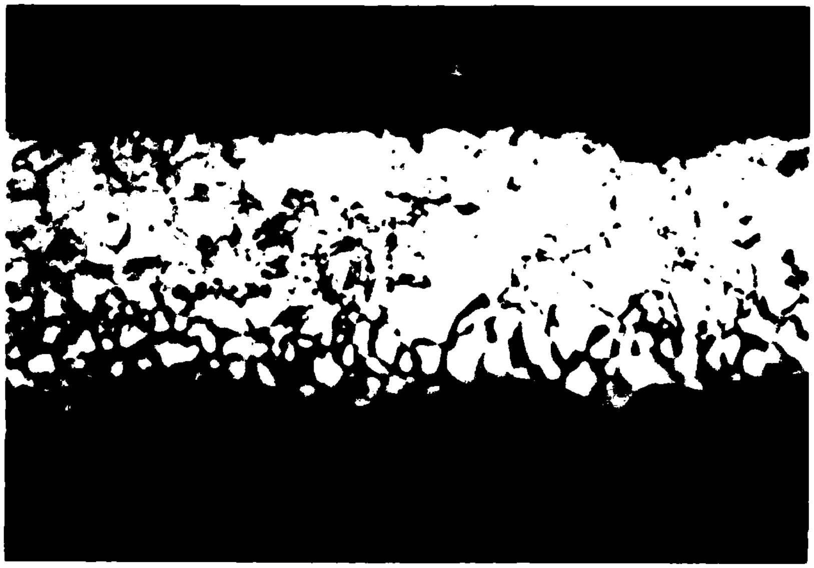
Figure 11. Extruded EZ33 Microstructure

x 25



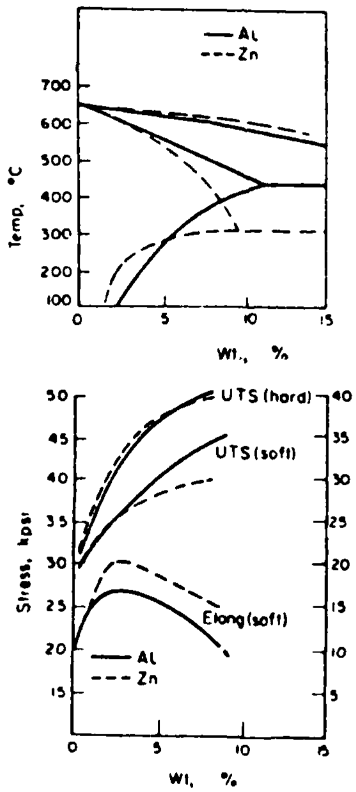
x 1000

Figure 12. Extruded EZ33 - Fracture Surface



x 1000

Figure 13. Mg 5.3% Zn Melt-Spun Ribbon



Effect of aluminium and zinc additions on the strength and ductility of rolled magnesium. Percentage elongation scale on right (after McDonald)

Figure 14.

Two extrusions were obtained and mechanically tested. Results are given in Table VIII. A reduction of the extrusion temperature from 450 to 310°C was obtained by using a more powerful press on the same die. The microstructure of the first extrusion, given Figure 15, displays a fine second phase distribution, but also the presence of intensive grain growth after recrystallization. Mechanical properties are therefore poor (Table VIII).

The second sample's microstructure, given in Figure 16, shows that the temperature was low enough to avoid grain growth, but not for recrystallization not to take place. A fine equiaxed grain structure is visible in both transverse and longitudinal directions. A comparison of mechanical properties given in Table VIII with those of rolled binary run R.S.P. samples given in Figure 14 show no marked improvement in the R.S.P. sample. 310°C is therefore still too high an extrusion temperature.

D. ZK60

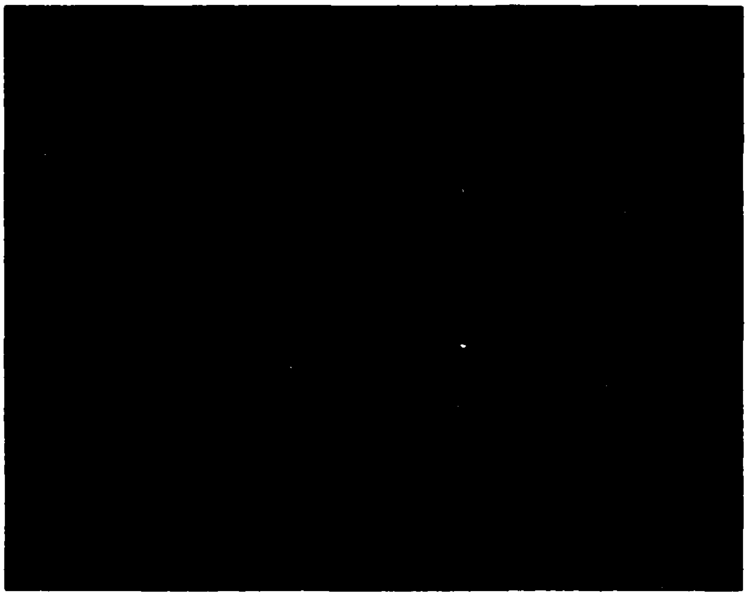
This alloy is a standard commercial grain refined wrought alloy. It is also one that has been investigated before, using R.S.P. techniques and extrusion procedures.

Microstructures of the melt-spun ribbons are given in Figure 17. As can be seen, no grain refinement is visible close to the chill, and throughout most of the 50 um ribbon thickness. Comparison with a piece of splatter (a 1-2 mm droplet) from the crucible during one of the melt-spinning runs, Figure 18, shows, however, that the grain refining action of the Zr addition had not disappeared during meltdown. Also, a thicker ribbon does display such a microstructure far from the chill, Figure 19. There is, therefore, a critical cooling rate, over which zirconium isn't active as a grain refiner in magnesium, and must, therefore, be either in solid solution, or in a very finely distributed precipitate.

TABLE VIII

Magnesium-Zinc Extrusion

Extrusion Ratio	1/28.4	1/28.4
Extrusion Pressure	22 ksi	50 ksi
Extrusion Temperature	450°C 20°C	310°C 10°C
Yield Strength	27.3 ksi	35.2 ksi
U.T.S.	40 ksi	46 ksi
Elongation	11%	25.4%



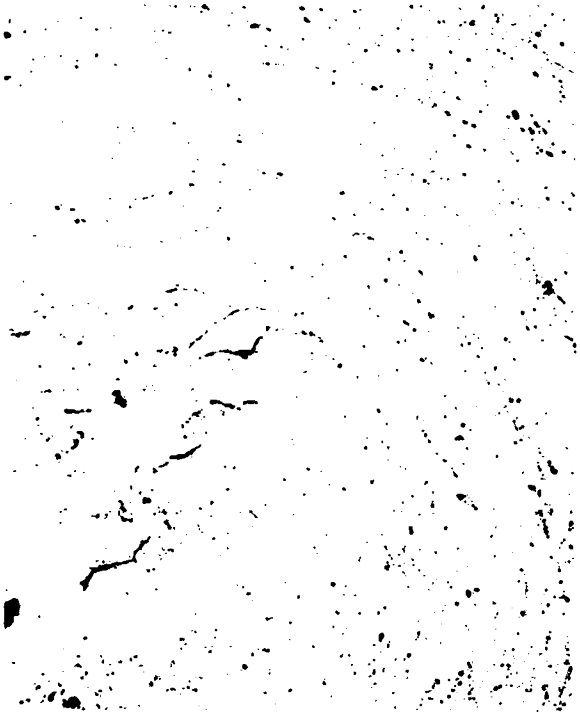
x 500

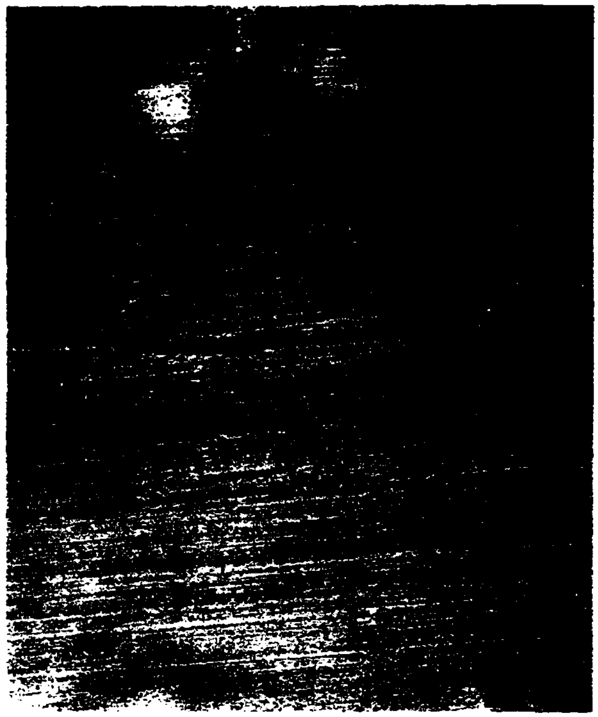
Longitudinal Cut

x 500

Transverse Cut

Figure 15 - Mg-5.[illegible]% Zn, extruded at 450°C.





x 50 x 50

Transverse Cuts x 1000 Longitudinal Cuts x 1000

Figure 16. Mg-5.3% Zn, Extruded at 310°C

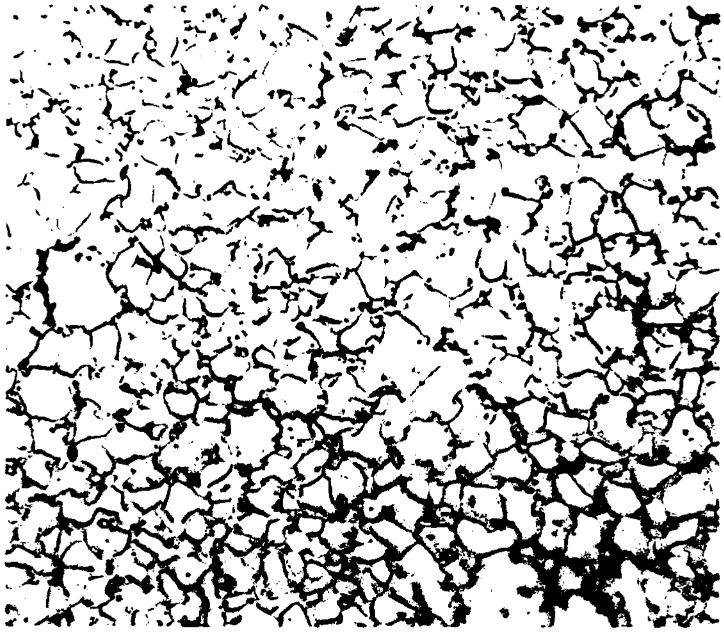
x 1000

Longitudinal Cut

x 1000

Transverse Cut

Figure 17. Melt-Spun ZK60



x 200

Figure 13. Splatter of ZK60

[illegible]

Two extrusions were obtained from these ribbons. One was extruded using the 10 ton press, and a 3/16 inch die. Properties are given in Table IX, and microstructures in Figure 20. The grain size is small, and although recrystallization has apparently taken place, grain growth was inhibited by the zirconium addition, as is usually the case[20]. The second phase distribution is not uniform, and on the same scale as the ribbon thickness, probably due to the lack of uniformity within the ribbons themselves.

Mechanical properties were slightly, but not very much, better than those of the commercial as received alloy, given in Table IX. Therefore, the extrusion temperature was substantially lowered, by means of a double extrusion procedure in the second sample.

A die, 0.41 inches in diameter was machined, and the green compact extruded once under the 20 ton press. The temperature was 210°C. The extruded sample was then cut, inserted into a pure magnesium sleeve, and the resulting composite was re-extruded at a similar temperature, using the same die. The total reduction in area ratio was therefore 1/35.4. The procedure is described in Figure 3. The resulting sample was machined down to a tensile specimen 0.100 inches in diameter, and was thus free of the surrounding Mg sheath over the gage length.

Mechanical properties are given in Table IX. The sample displayed a nearly perfect textbook elastic-plastic curve and properties compare well with those obtained by Isserow and Rizzitano[13].

Microstructures are given in Figure 21 and 22. After the first extrusion, Figure 21, a banded structure can be discerned,

20. E.F. Emley, Supra note 2, page 506.

TABLE IX

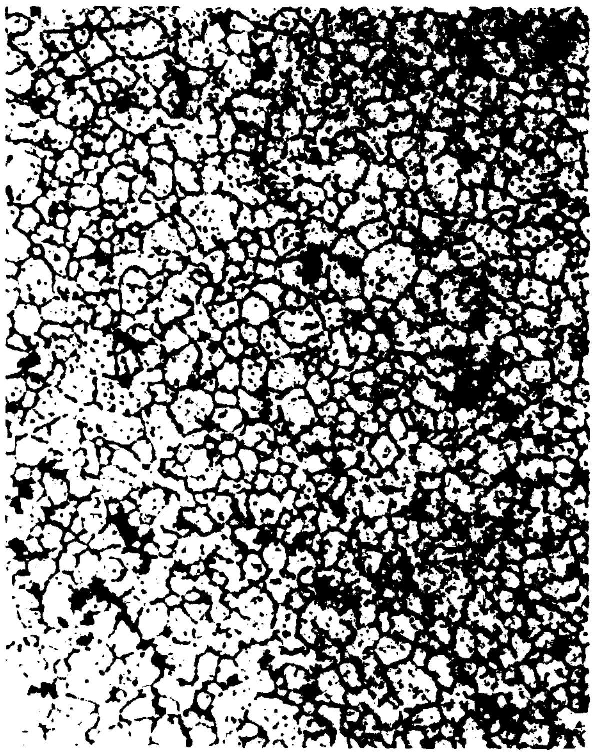
ZK60 Extrusions

	ZK60A-T5 Extruded Rods (Metals Handbook)		First Extrusion	Second Extrusion
Extrusion Ratio		1/27.4	1/6	1/6
Extrusion Pressure		22 ksi	50 ksi	25 ksi
Extrusion Temperature		470°C 20°C	210°C 15°C	210°C 20'
Yield Strength	41 ksi	32.6 ksi		53.0 ksi
U.T.S.	51 ksi	47.3 ksi		56.3 ksi
Elongation	11%	21.1%		19.6%

x 100

x 1000

Longitudinal Cuts

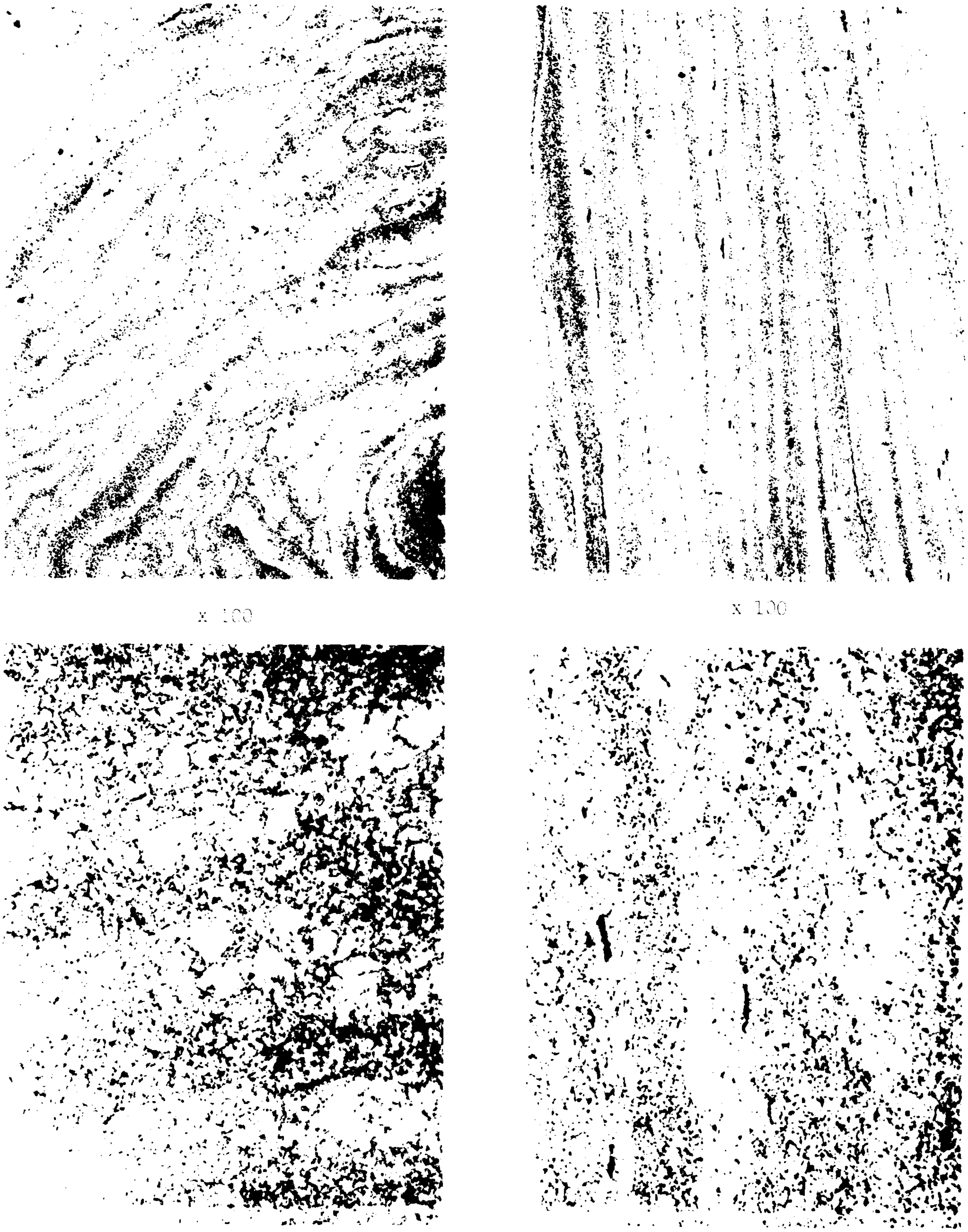


Transverse Cuts

x 100

x 1000

Figure 20. ZK60 Extruded at 470°C



Transverse Cuts x 1000

Longitudinal Cuts x 1000

Figure 11. EX60 - First Extrusion at 210°C

on the scale of the extruded ribbons. The white grains, therefore probably correspond to the white "Featureless" zones displayed by the ribbons. Oxide films are clearly visible, and this sample displayed some delamination at the tip when re-extruded. The reduction in area ratio of 1/6 was not sufficient for full densification.

The re-extruded microstructure, Figure 22, is finer still, and the white bands less easily discernable, but still present. Throughout both extrusions, no noticeable amounts of recrystallization are apparent.

The fracture surfaces are given in Figure 23. A very slight delamination is still discernable to a critical eye, but the fracture surface is ductile, and is completely different from that observed by Isserow and Rizzitano[13]. It is therefore believed that this product would have superior transverse properties.

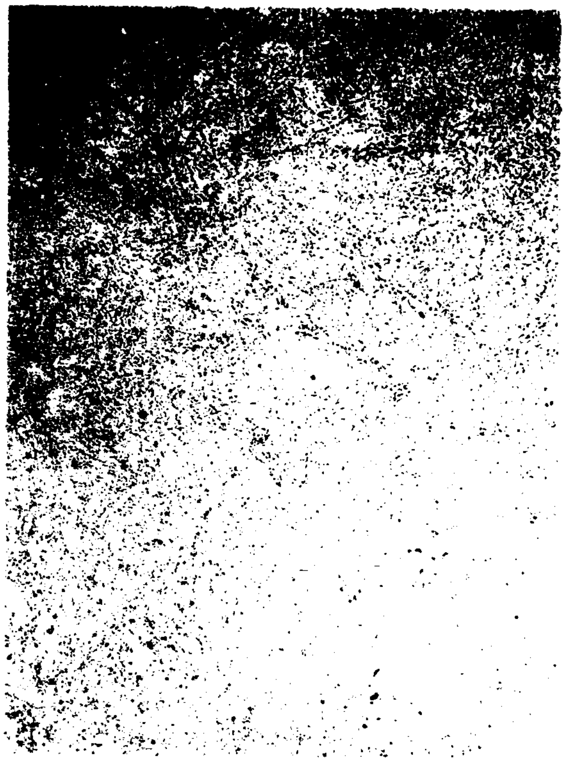
IV. CONCLUSIONS

1. A melt spinning apparatus was constructed at MIT, with a relatively large crucible to allow for rapid obtention of ribbons for consolidation purposes.

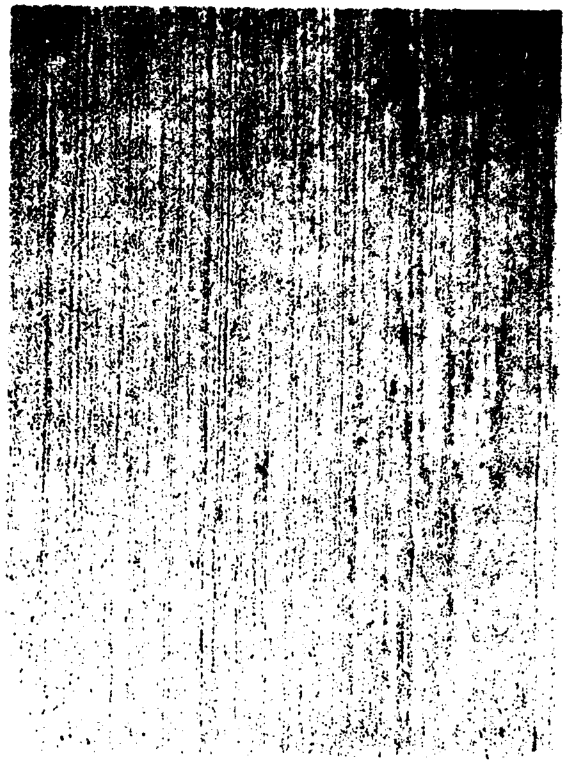
2. Casting under a controlled atmosphere is advantageous in avoiding the risk of burning inherent to molten and finely divided magnesium.

3. Zinc containing magnesium alloys wet iron very well. This may lead to instabilities in the metal stream if no precautions are taken.

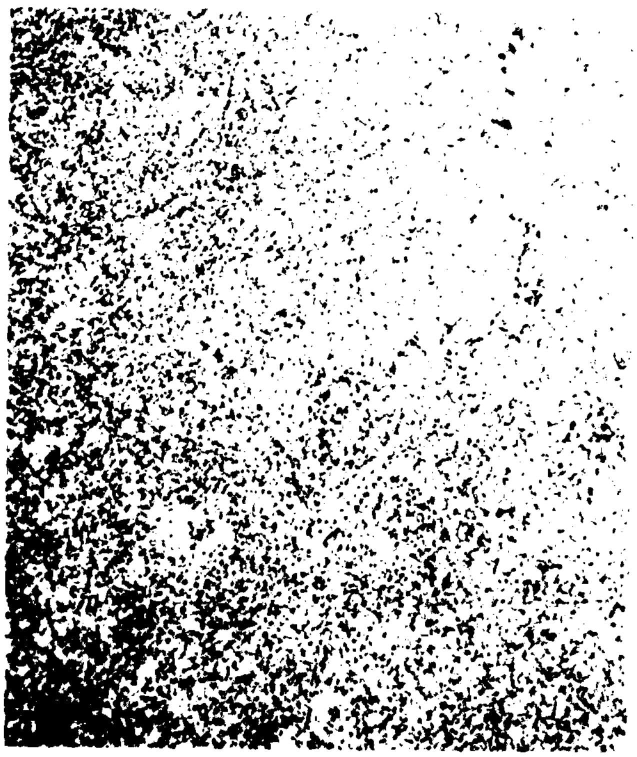
4. The existence of a "featureless" zone in the ribbons is



x 100



x 100



Transverse Cuts x 1000

Longitudinal Cuts x 1000

Figure 22. ZK60 - Second Extrusion at 210°C

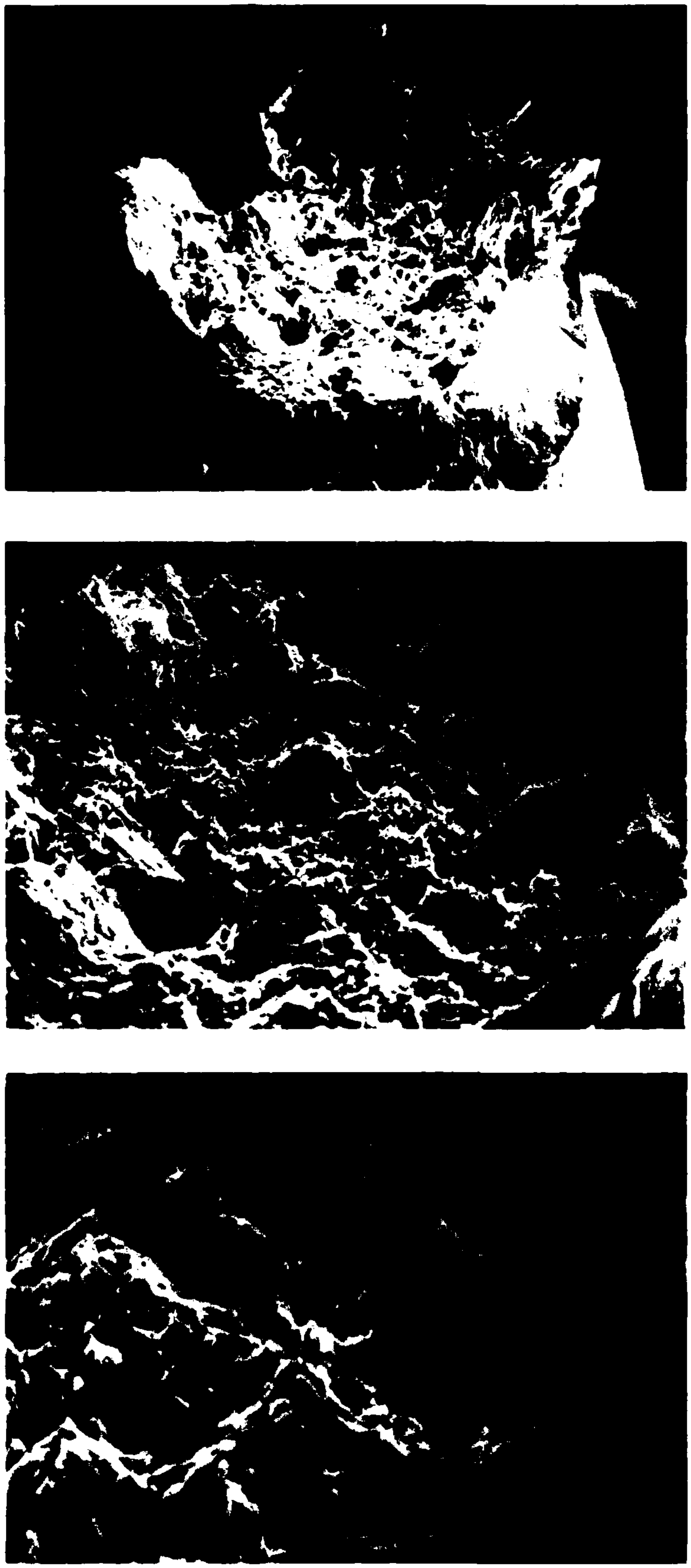


Figure 23. ZK60 Double Extrusion at 210°C - Fracture Surface

proved. It is located along the chill face, and its fraction increases as the ribbon thickness decreases. In particular, the grain refining action of zirconium is suppressed in that zone.

5. Hot extrusion conditions have been studied in a laboratory bench scale for these R.S.P. magnesium alloys.

6. Full, delamination free, densification is obtained with a reduction in area ratio of about 1/30.

7. The extrusion temperature is of paramount importance in obtaining the remarkable properties of rapidly solidification materials for magnesium alloys. Samples of Mg-5.3%Zn, EZ33, ZK60 extruded above 300°C gave non-exceptional properties. The metal recrystallizes, and in non-zirconium containing alloys, grain growth occurs at higher temperatures. Also, the second phase can precipitate out, as occurred in EZ33.

8. To produce interesting properties in the extruded R.S.P. sample, temperatures lower than about 250°C are necessary. This correlates well with previous data, as exposed in the introduction.

9. A double extrusion procedure was designed to extrude ZK60 at these temperatures.

10. Using these apparati, techniques, and conditions, the following properties were obtained in ZK60: Y.S. 53.0 ksi
U.T.S. 56.3 ksi
Elongation: 19.6%
Y.S. 41 ksi
U.T.S. 51 ksi
Elongation: 11%

for conventionally extruded ZK60. The fracture surface proved that a fully dense, delamination free sample was obtained.

It is thus proved that Rapid Solidification can yield remarkable materials when applied to magnesium alloys. This sample was not heat treated, was not canned, and the whole process has, still, margin for improvement. The alloy compositions can be varied, and the materials further characterized. This should give an idea of the potential lying in this barely explored field, the Rapid Solidification Processing of magnesium alloys.

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